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Standard Guide for Measurement of Gas Permeability and Gas Diffusivity of Carbon and Graphite[1]

This standard is issued under the fixed designation D8563; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the measurement of gas permeability and gas diffusivity of carbons and graphite. Two techniques are discussed, the static equilibrium method, an integrated method capable of measuring both gas permeability and gas diffusivity, and the vacuum leak method, which measures the gas permeability of the specimen.

1.2 Both of the gas permeability techniques discussed assume laminar flow through the pores of the specimen. For the purposes of analysis, it is assumed that the resultant gas permeability consists of viscous flow (pressure driven gas flow) and slip (the interaction of gas molecules with the walls of the pores). Generally, the contribution from slip is much smaller than that from viscous flow but can be significant in cases of low permeable flow and specimens with very fine pore sizes.

1.3 Gas permeability measurements are discussed for the vacuum leak method at mean pressures up to 50 kPa and differential pressures in the range 5 kPa to 50 kPa.

1.4 Gas permeability measurements are discussed for the static equilibrium method at a mean pressure of 100 kPa and differential pressures in the range of 0.01 kPa to 0.15 kPa. Gas diffusivity measurements are also discussed for the static equilibrium method under the same conditions detailed in 1.4.

1.5 This guide is based on experiments conducted on small specimens between 12Ø × 20 mm and 19Ø × 55 mm, but the test methods may be suitable for other sizes. Should the testing of specimens outside of this geometry be required, the user will need to undertake testing to confirm the techniques are suitable. Viscous coefficients in the range from 0.5×10^{-10} cm^2 to 200.0×10^{-10} cm^2 are readily measurable by the discussed test methods. Gas diffusivity ratios in the range of 0.001 to 0.5 are readily measurable by the static equilibrium method. The measurements and analysis from which this guide has been derived have been published as an ASTM technical paper.[2]

1.6 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

NOTE 1—The term permeability as used in this guide considers gas flow where both viscous and slip components are present, resulting in units of cm^2 s^{-1}. For comparison with sources where permeability is given in units of cm^2, readers should consider the viscous coefficient, B_0, as these are equivalent to the Darcian permeability when slip contributions are negligible or otherwise accounted for.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4525 Test Method for Permeability of Rocks by Flowing Air (Withdrawn 2022)[4]

3. Terminology

3.1 *Definitions:*

[1] This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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[2] J. H. Dinsdale-Potter, D. Charlton, T. Shaw, and A. Tzelepi, "Measurement of Gas Permeability of Irradiated and Virgin Graphite Specimens," in *Graphite Testing for Nuclear Applications: The Validity and Extension of Test Methods for Material Exposed to Operating Reactor Environments*, ed. A. Tzelepi and M. Metcalfe (West Conshohocken, PA: ASTM International, 2022), 130–159.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

3.1.1 For definitions of terms used in this guide, refer to Terminology D4175.

3.1.2 *gas diffusion coefficient, n*—a measure of the gas molecular flux that occurs for a given concentration gradient.

3.1.3 *gas diffusivity, n*—a measure of the amount of gas that will diffuse through a material in the absence of a pressure gradient, with gas flow driven by concentration gradients.

3.1.4 *gas diffusivity ratio, n*—the ratio of gas diffusivity in the specimen against the free diffusion of gases in air.

3.1.5 *gas flux, n*—the amount of gas passing through a given area for a given period of time.

3.1.6 *gas permeability, n*—a measure of the amount of gas that will pass through a material in the presence of a pressure gradient across the material.

3.1.7 *parts per million, n*—all reference to parts per million (ppm) are by volume.

3.1.8 *slip coefficient, n*—a component of gas permeability which represents gas transport that occurs due to 'slip', the interaction of gas molecules with the walls of the pores of a material.

3.1.9 *viscous coefficient, n*—a component of gas permeability which represents the pressure driven component of permeable gas flow through a material.

4. Static Equilibrium Method for Gas Permeability and Gas Diffusivity – Summary

4.1 Two different gases are passed through the specimen in flow and counter-flow, with the specimen sealed such that gas can only pass through the specimen. The effective differential pressure is then controlled by varying the flow rates of each gas, with the differential pressure measured using a manometer in parallel with the gas flow through the specimen. The concentrations of the gases are measured on the opposite side of the specimen from which they enter, from which the gas molecular flux for each gas can be calculated.

4.2 Maintaining both gases at equal pressure, that is, with no pressure gradient across the specimen, allows for the gas diffusivity to be measured.

4.3 By varying the applied differential pressure and measuring the resultant change in fluxes, the gas permeability of the specimen can be calculated. The viscous and slip permeability coefficients cannot be determined by this method; hence it is only applicable to materials with negligible slip contributions.

4.4 The critical factors in this method are:

4.4.1 Gas leaks to or from atmosphere prior to transport through the specimen and measurement of gas concentrations must be minimized to below detectable levels.

4.4.2 All gas flow through all components of the measurement equipment must be steady-state laminar flow.

4.4.3 Differential pressure across the specimen should never exceed that which prevents the counter flow of the opposing gas.

4.4.4 The other conditions such as the average flow rate, specific method of sealing, and purge gas composition are less critical, and the experimenter should determine the sensitivity of the gas permeability measurements to these conditions.

5. Vacuum Leak Method for Gas Permeability – Summary

5.1 Two pressure tanks are connected by the specimen which is sealed to only allow gas to enter and exit from opposite faces. A steady flow of air is provided into the initial tank, and the second tank is drained to vacuum. Both tanks are maintained at constant pressure as the gas flows through the specimen into the second tank. The exhaust to vacuum on the second tank is then sealed and the rise in pressure in the second tank is measured over a set period of time. With known sizes of the tanks, the effective rate of gas flow through the specimen, and hence gas permeability of the specimen can be calculated. By varying the mean pressure across the two pressure tanks the viscous and slip permeability coefficients can be determined.

5.2 The critical factors in this method are:

5.2.1 Gas leaks to or from atmosphere prior to transport through the specimen and into the outlet pressure tank should be minimized to below detectable levels.

5.2.2 All gas flow through all components of the measurement equipment must be steady-state laminar flow.

5.2.3 Differential pressure across the specimen should be significantly larger (by at least a factor of 5) than the pressure of the lower pressure outlet tank.

5.2.4 The other conditions, such as the specific gas used for the measurement or the method of sealing, are less critical, and the experimenter should determine the sensitivity of gas permeability measurements to these conditions.

6. Significance and Use

6.1 Gas permeability and gas diffusivity are important properties required for correctly modeling and understanding interactions between gases and porous media, for example thermal and radiolytic oxidation, or the escape of gaseous fission by-products through graphite.

6.2 The test methods described in this guide can be used to characterize graphite and carbon materials for design purposes and post irradiation examination (PIE).

7. Test Specimen

7.1 Test specimen considerations are the same for both the vacuum leak method and the static equilibrium method.

7.2 A typical specimen is a cylinder, between 20 mm and 40 mm in length and 12 mm and 20 mm in diameter; no definitive optimal geometry has been determined for these techniques, but other, similar methods suggest specimens with a length to diameter ratio in the range of 1.3 to 1.7 (Test Method D4525). Limited studies on specimen geometry for these techniques do not suggest an explicit size dependence[2]; however, the resultant permeable and diffusive flows are inversely proportional to the ratio of length to cross sectional area. Specimens which are comparatively too long will be difficult to accurately measure due to the low resultant flow. Conversely, specimens which are comparatively too short risk leaving the laminar flow regime, for which the calculations detailed in later sections are not applicable. Although no direct study has been made on the limitations imposed by grain size